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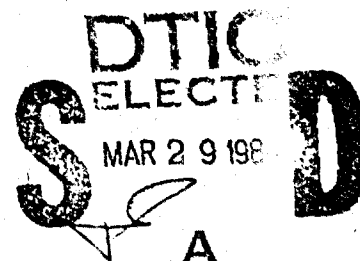
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CONTRACTOR REPORT ARLCD-CR-83054

## COMBUSTIBLE CARTRIDGE CASE CHARACTERIZATION

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FEBRUARY 1984



**U.S. ARMY ARMAMENT RESEARCH AND DEVELOPMENT CENTER**  
**LARGE CALIBER WEAPON SYSTEMS LABORATORY**  
**DOVER, NEW JERSEY**

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The beater additive process for manufacturing combustible cartridge cases is superior to the solvent resin, post-impregnation process. However, the current beater additive resins do not produce as good a product as the solvent-resin system. The objective of this program, therefore, was to determine if either or both of two new beater additive resin systems could produce combustible case material equal to that produced by the post-impregnation process. (cont)		

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19. KEY WORDS (cont)

Kor-Lok 42-3001  
Cymel melamine resin  
Akaradit II stabilizer

20. ABSTRACT (cont)

Test coupons of combustible cartridge case material were fabricated using these recommended resins. The procedure was similar to that used in current commercial production runs. The tensile strengths of these coupons were similar to the strengths obtained with coupons prepared with the current commercial resin.

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## INTRODUCTION

The beater additive process for manufacturing combustible cartridge cases is superior to the solvent-resin post-impregnation process. However, the current beater additive resins do not produce as good a product as the solvent-resin system. The objective of this program, therefore, was to determine if either or both of two new beater additive resin systems could produce combustible case material equal to that produced by the post-impregnation process. Samples of case material were to be prepared in a commercially compatible manner.

A recent contract (DAAK10-79-C-0074) at New York University (NYU) has resulted in the selection of two cross-linked melamine/formaldehyde acrylic styrene resin systems that can be used in the beater additive process. The resin systems are Dow Chemical Co. latex No. 241 and National Starch and Chemical Co. resin No. 78-3730. The NYU study showed that each of these systems could be significantly improved by the addition of a cross-linking agent, Cymel 300, manufactured by American Cyanamid Co.

## EXPERIMENTAL PROCEDURES

Proper evaluation of combustible cartridge material depends on many factors: the composition of the test specimens must be firmly established; the procedures used to prepare test specimens must be consistent; all variations must be recorded; and instruments must be frequently calibrated and test equipment checked for accuracy. The methods used to ensure valid data on this program are described below.

### MATERIAL HANDLING

#### Nitrocellulose

Each drum of nitrocellulose fibers was labelled with the nitrogen content of the fibers and the approximate water content of the drum. Fibers with a nitrogen content of 12.6% were used in all batches except 1, 2 and 4. When required, portions of nitrocellulose fiber were removed from the drum and held in sealed plastic bags. The moisture content of each bag was determined experimentally by analyzing small representative samples. The amount of wet fibers to be used to provide the desired amount of dry material for a batch could then be calculated.

#### Kraft Fibers

The Kraft fibers were received as sheets weighing approximately 1.3 lb each. The moisture content of the sheets varied from 6 to 7-1/2 percent, depending on the relative humidity of the laboratory. The moisture content was checked regularly to ensure that the desired percentage of Kraft fibers was incorporated into each batch.

#### Resins

The resins were supplied as a water emulsion with nominally 50 percent solids. Samples of each container were analyzed and the exact percent solids determined. Cymel 300 resin, a waxy solid at room temperature, contains 98 percent solids. Prior to use it was dissolved in a methanol-water solution.

### Akaradit II

Akaradit II (N-methyl-N'-diphenyl-urea) serves as a stabilizer for the nitrocellulose. One weight percent of this finely ground powder was added to each batch.

### Lufax 295

As a retention aid for the resins, 0.05 percent Lufax 295 was added to all batches. A 1 percent stock solution was prepared and used to measure the correct quantity for each batch accurately.

### Alum

Papermakers' alum, aluminum sulfate, was obtained from Fisher Scientific Co. A stock solution of 10 percent solids in water was prepared.

### Miscellaneous Chemicals

Catalysts and other chemicals were acquired as needed. Stock solutions in water were prepared to eliminate handling and measuring problems.

### Mass Balance

After all test coupons of a batch had been felted, the remaining solids in the felting tank were collected, dried, and weighed. The total weight of the test coupons plus this residual weight should equal the amount of solids used in preparing the batch. Mass balances of 98-99 percent were usually obtained for each batch.

## **SPECIMEN PREPARATION**

### Slurry Preparation

Each batch was prepared with sufficient material to produce 50 test specimens. A standard preparation procedure was developed for all batches. Some variations in the procedure did occur and are noted in Section 3. The standard procedure is outlined below.

- The nitrocellulose and Kraft fibers are slurried in water at a 2.0 percent solids content
- Lufax 295, at 0.05 wt % of the fibers, is added and agitation continued for 30 min
- The resin emulsion is added over a 5-min period and agitation is continued for another 30 min

- Akaradit II is introduced into the slurry
- Papermakers' alum is added over a 30-min period to reduce the pH to 4.4-4.5
- The mix is allowed to age overnight
- The contents of the stock tank are divided into aliquots equal to one felt sample weight.

#### Felting Operation

The felting equipment consisted of a 50 gal felting tank, a calibrated vacuum tank, and the necessary auxiliary equipment and plumbing. The felting tank contained a slurry with a solids content of about 0.15 percent. The amount of material felted was determined by drawing a predetermined amount of water through the felting die. A double-headed felting die delivered two wet forms, each 1-3/4 in. wide by 7 in. long. Squeezing on the felting die reduced the water content to about 60 percent. The vacuum on the felting die was broken with compressed air and the wet forms removed. The white water was then returned to the felting tank and an aliquot of solids equal to the felted specimens was dispersed in the felting tank. The tank was now ready for the next felting operation. The pH of the felting tank could be controlled or lowered by adding alum or other chemicals.

#### Pressing and Curing

The wet forms were placed in aluminum molds and pressed to the proper thickness between heated platens. The temperature of the platens was usually 250°F; however, temperatures as low as 220°F and as high as 300°F were investigated. The coupons were held in the heated molds from 2 to 16 min. Tests with thermocouples inserted into the coupons showed that with a press temperature of 250°F, the coupon reached 230°F in 8 min. Upon removal from the hot press, each coupon was given the appropriate sample number and lot number. Final drying and resin curing was completed by drying overnight in an oven maintained at 150°F.

#### Coupon Identification

The coupons were removed from the oven in the morning and their weight recorded. The thickness was measured at several points and the average recorded. Density was calculated from the weight of the coupon and its dimensions. Two coupons were cut into 1/2 x 1-3/4 in. segments and densities were determined for each segment. The density of the coupons was found to be

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consistent to within 1 percent along the 7-in. length. Variations in density within one lot did occur because of variations in felted weight. Different resin systems shrink differently upon curing causing variations in densities between batches.

### Physical Testing

Coupons were selected from each lot for tensile testing by ASTM method D638-64T. Six-inch "dog bone" test specimens were prepared by cutting out the center of the coupon with an appropriately shaped steel die (Figure 1). Before testing, the specimens were held for 24 hr in the testing room, which was maintained at 73°F and 50 percent relative humidity. The tests were conducted on an Instron Engineering Corp. Model TTB tensile tester. The test specimens were clamped with a 4.5 in. starting distance between machine grips; the cross-head speed was 0.1 in. per minute.

The stress/strain curve was recorded on a Leeds and Northrup Speedmax recorder. A typical record is presented in Figure 2. The tensile strength is calculated by dividing the maximum load in pounds by the original minimum cross-sectional area in square inches. The result, in pounds per square inch, can be reported to three significant figures.

The stress/strain curve was examined to determine the amount of specimen extension at the moment of rupture. The percentage elongation was calculated by dividing the extension by 2.25 in. (the original length of the test section) and multiplying by 100. The result is reported to two significant figures.

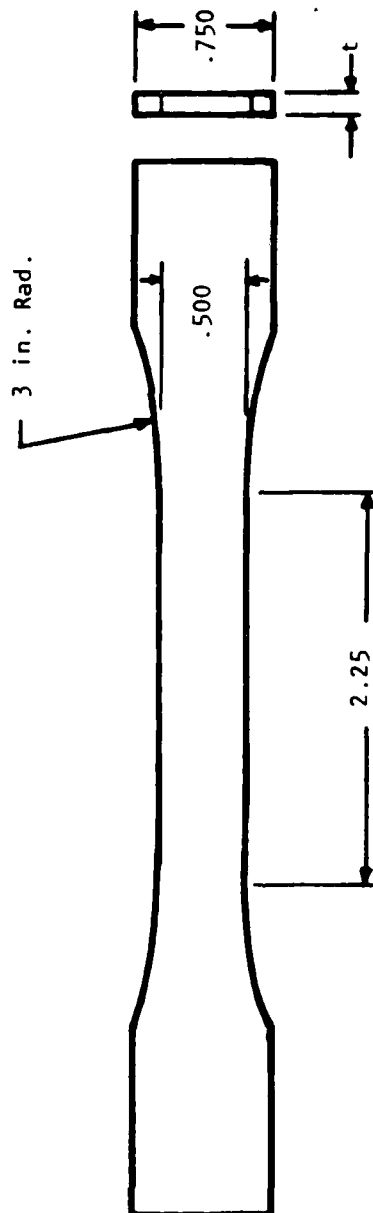


Figure 1. Physical strength tension test specimen.

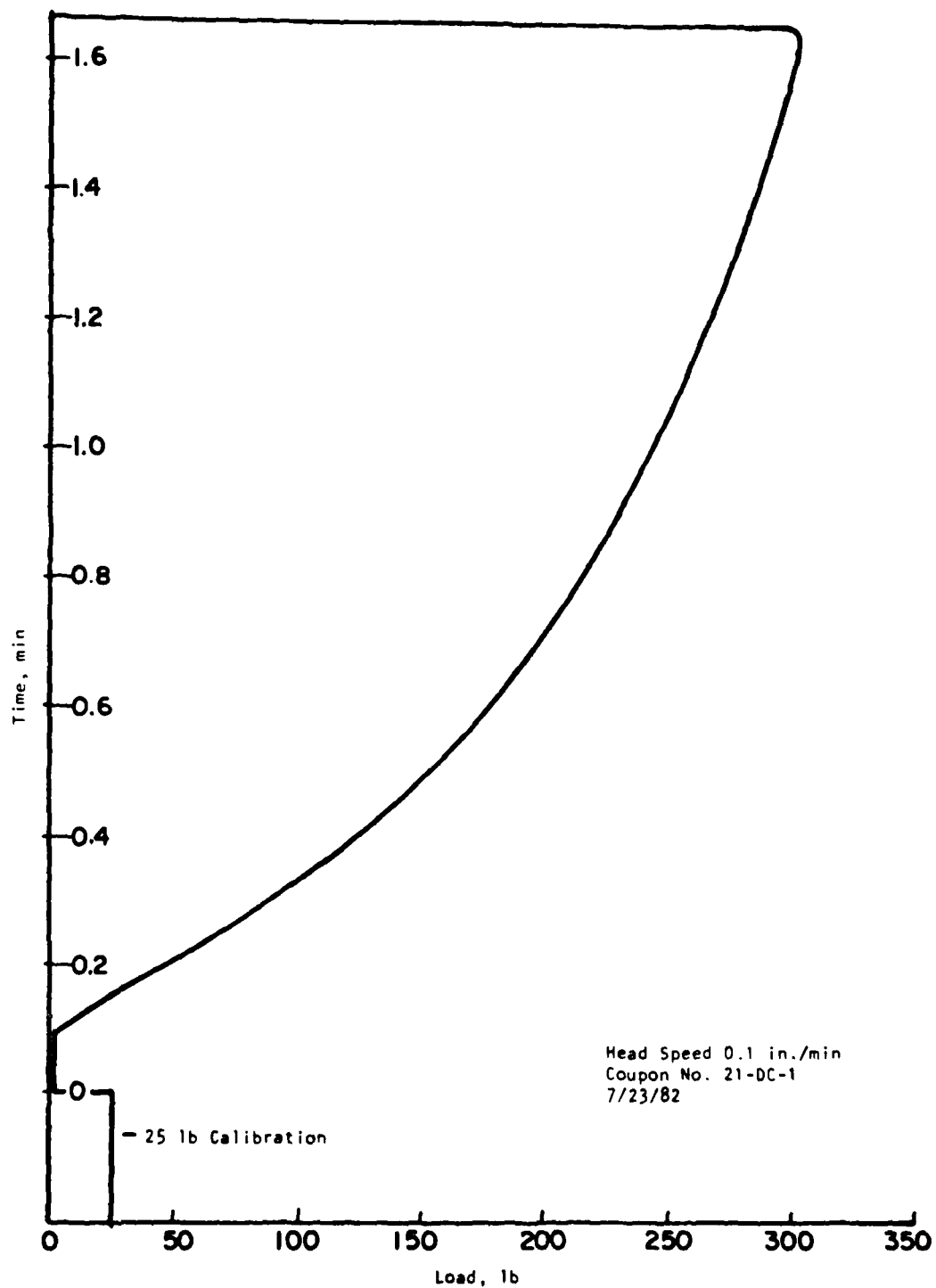


Figure 2. Typical stress-strain curve.

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## EXPERIMENTAL RESULTS

### DATA COLLECTION AND REDUCTION

Approximately 50 test coupons were obtained from each lot. Groups of coupons were pressed at various temperatures and for various time periods to determine the effect of these parameters. The data from each lot were plotted to show the tensile strength as a function of density. Other variables, such as press time and temperature, were identified. Typical graphs are shown in Figures 3 and 4.

Figure 3 indicates that tensile strength can be considered a linear function of the density in the small density range under consideration. This observation was common to all lots and was very helpful in interpolating/extrapolating the data to a common reference density--0.875 g/cc. For this lot, the tensile strength at 0.875 g/cc was 4000 psi. The data indicate that at a press temperature of 250°F, doubling the period of pressing from 8 to 16 min had no effect. Coupons dried and then pressed at 250°F had a lower density, because of spring back, but the extrapolated tensile strength was the same as that of the other coupons. Specimens that were aged 3 days before pressing still had the same strength. Those specimens that were felted and pressed at a lower pH (4.5) were noticeably weaker.

Thirty-two slurries were prepared. Thirty-one were successfully felted and representative coupons tested for tensile strength. Table 1 summarizes the data obtained in these runs.

### VARIATIONS IN PROCEDURE

The data of Table 1 show that Cymel 300 was not contributing to the tensile strength of the coupons. The preparation procedure, the felting procedure, or the pressing conditions probably had to be modified to gain the expected benefits of the cross-linking agent.

Our simulation of the conventional commercial method of producing cartridge cases was reviewed and considered satisfactory with one exception. In the laboratory preparation of aliquots, the contents of the 15-gal

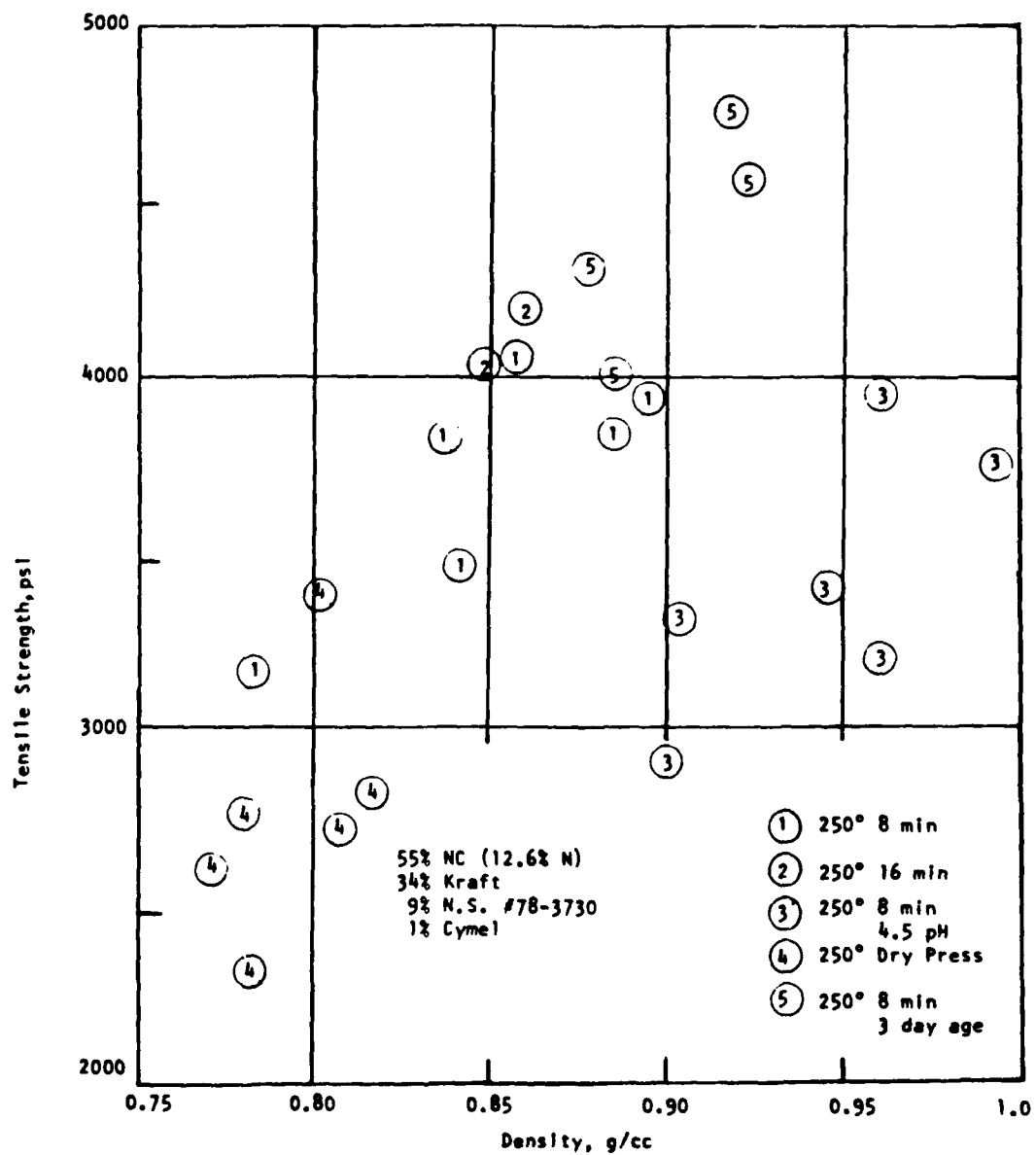


Figure 3. Density-tensile strength relationship for Lot No. 12NC.

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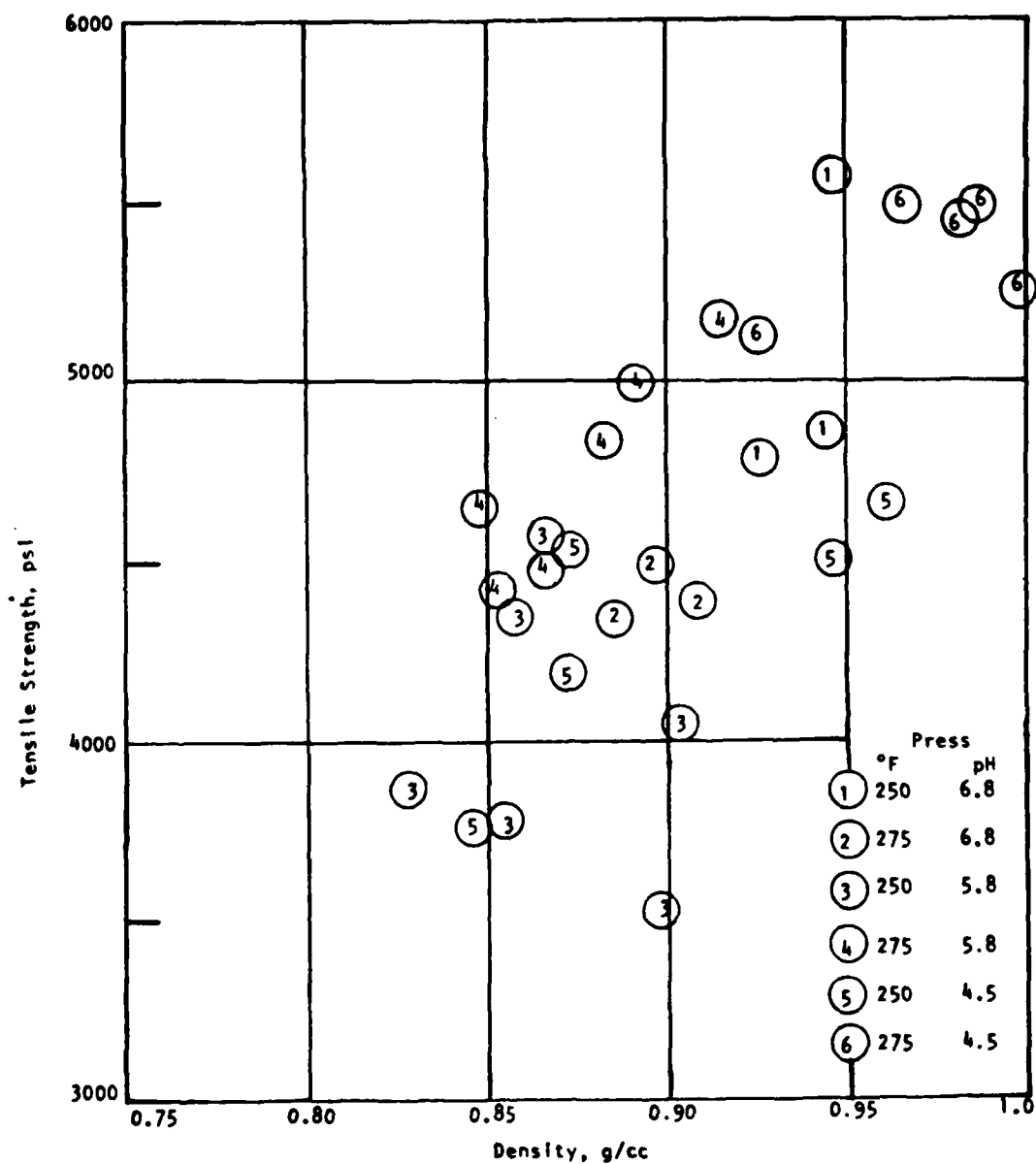


Figure 4. Density-tensile strength relationship for Lot No. 19DC.  
(55% NC, 34% Kraft, 10% Dow 241, 0.2% Cymel 300)

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TABLE 1. DATA SUMMARY (page 1 of 3)

Lot No.	Composition, wt %			pH		Press		Tensile Strength, psi	% Elongation
	Fibers		Resin System	Prep	Felt	Temp., °F	Time, min		
	WC	Kraft							
1	72 <sup>1</sup>	17	10 Dow 241	4.5	7.4	220	8	2300	6.0
2	72 <sup>1</sup>	17	10 N.S. 78-3730**	4.5	7.4	220	8	2600	9.3
3	72	17	10 Dow 241	4.5	7.4	250	8	3200	7.0
4	72 <sup>1</sup>	17	5 Dow 241 5 Cymel 300	4.5	7.4	220	8	2000	7.5
				4.4	7.4	300	8	2600	8.0
				4.4	7.4	250	8	2600	8.0
				4.4	7.4	220	8	1800	7.7
5	55	34	5 Dow 241 5 Cymel 300	4.5	7.4	250	8	3700	7.5
				4.5	7.4	280	6	3700	7.5
				4.5	7.4	250	4	3200	8.6
6	55	34	5 N.S. 78-3730 5 Cymel 300	4.5	7.4	250	8	3500	8.0
				4.5	7.4	250	16	3500	8.0
7	55	34	7.5 N.S. 78-3730 2.5 Cymel 300	4.5	7.4	250	8	4500	9.0
8	55	34	10 Dow 241	4.5	7.4	250	8	4500	8.5
				4.5	7.4	250	16	4500	8.5
9	55	34	7.5 Dow 241 2.5 Cymel 300	4.5	7.4	250	8	4500	7.5
10	55	34	10 N.S. 78-3730	4.5	7.4	250	8	4700	8.0
				4.5	7.4	250	16	4700	8.5
11	55	34	9 Dow 241 1 Cymel 300	4.5	7.4	250	8	4300	9.0
12	55	34	9 N.S. 78-3730 1 Cymel 300	4.5	7.4	250	8	4000	11.2
				4.5	7.4	250	16	4000	11.2
				4.5	7.4	250	8	4000	11.2 <sup>2</sup>
				4.5	4.5	250	8	2800	11.2
13	55	34	9 Dow 241 1 Cymel 300	4.5	7.4	250	8	4300	8.2 <sup>3</sup>
14	55	34	10 Cymel 300	3.0	3.0	250	8	1600	5.5
				3.0	3.0	300	8	1600	4.4
				3.0	3.0	275	8	1400	4.0 <sup>2</sup>
15	53	34	10 Dow 241 + 0.5 Cymel 300	3.0	3.0	250	8	4300	8.0
				3.0	3.0	250	16	4100	6.9
				3.0	3.0	300	8	2600	3.1
				3.0	3.0	300	16	2600	2.9
16	55	34	10 N.S. 78-3730 + 0.5 Cymel 300	3.0	3.0	250	8	3700	10.4
				3.0	3.0	250	16	3700	8.9
				3.0	3.0	275	8	4500	8.6
				3.0	3.0	275	16	3400	5.3
				3.0	3.0	300	4	4200	9.1
				3.0	3.0	300	8	2700	5.1

\* NC = nitrocellulose

\*\* N.S. = National Starch and Chemical

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TABLE 1. DATA SUMMARY (page 2 of 3)

Lot No.	Composition, wt %			pH		Press		Tensile Strength, psi	% Elongation
	NC	Fibers Kraft	Resin System	Prep	Felt	Temp., °F	Time, min		
17	55	34	10 Dow 241 + 0.5 Cymel 300	4.5	6.8	250	8	4600	9.0
				4.5	6.8	275	8	4500	9.5
				4.5	6.8	275	16	4600	9.8
				4.5	5.8	250	8	4800	9.0
				4.5	5.8	275	8	4800	9.0
				4.5	5.8	275	16	4800	8.7
				4.5	4.5	250	8	4900	8.9
				4.5	4.5	275	8	4900	9.0
				4.5	4.5	275	16	5000	8.9
				4.5	4.5	250	8	5000	7.0
18	0	77	10 Dow 241 + 0.5 Cymel 300 12% graphite yarn tensile strength at 0.75 g/cc	4.5	4.5	300	8	5800	5.3
				4.5	4.5	350	8	3800	3.1
				4.5	4.5	400	8	2500	1.3
				4.5	3.1	300	8	5800	4.1
				4.5	3.1	350	8	3300	1.8
				4.5	3.1	400	8	2500	2.0
				4.5	6.8	250	8	4200	8.0
				4.5	6.8	275	8	4300	7.5
19	55	34	10 Dow 241 + 0.2 Cymel 300	4.5	5.8	250	8	4400	9.2
				4.5	5.8	275	8	4700	9.1
				4.5	4.5	250	8	4200	8.5
				4.5	4.5	250	8	4200	8.2
				4.5	6.8	250	8	4200	7.6 <sup>a</sup>
				4.5	6.8	250	8	5000	8.2
				4.5	6.8	275	8	4200	8.8
				4.5	5.7	250	8	5100	8.6
20	55	34	10 Dow 241	4.5	5.7	275	8	5100	8.2
				4.5	4.5	250	8	5000	8.5
				4.5	4.5	275	8	5100	8.5
				4.5	6.6	250	8	5000	7.6
				4.5	6.6	275	8	5000	7.4
				4.5	5.4	250	8	5300	7.8
				4.5	5.4	275	8	5300	7.6
				4.5	4.4	250	8	5000	7.5
21	55	34	10 Dow 241 + 0.5 Cymel 300	4.5	4.4	275	8	4800	7.8
				4.5	6.8	250	8	4500	8.4
				4.5	6.8	275	8	4700	8.0
				4.5	5.5	250	8	4800	8.1
				4.5	5.5	275	8	4600	8.4
				4.5	4.5	250	8	4500	8.5
				4.5	4.5	275	8	4400	8.4
				4.5	4.5	275	8	4400	8.4
22	55	34	10 Dow 241 + 0.5 Cymel 300 ZnCl <sub>2</sub> as retention aid Zn(NO <sub>3</sub> ) <sub>2</sub> as catalyst	4.5	6.8	250	8	4500	8.4
				4.5	6.8	275	8	4700	8.0
				4.5	5.5	250	8	4800	8.1
				4.5	5.5	275	8	4600	8.4
				4.5	4.5	250	8	4500	8.5
				4.5	4.5	275	8	4400	8.4
				4.5	4.5	275	8	4400	8.4
				4.5	4.5	275	8	4400	8.4

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TABLE 1. DATA SUMMATION (page 3 of 3)

Lot No.	Composition, wt. %			pH		Press		Tensile Strength, psi	% Elongation
	Fibers	Kraft	Resin System	Prep	Felt	Temp., °F	Time, min		
23	55	34	10 Dow 241 + 0.5 Cymel 303	4.5	6.8	250	8	5400	7.8
				4.5	6.8	275	8	4500	7.6
				4.5	5.5	250	8	4600	7.4
				4.5	5.5	275	8	5500	8.0
				4.5	4.5	250	8	4700	8.0
				4.5	4.5	275	8	4800	7.5
24	55	34	10 Dow 241 + 0.5 Beetle	4.5	6.7	250	8	3900	9.7
				4.5	6.7	275	8	5000	9.0
				4.5	5.5	250	8	4600	8.0
				4.5	5.5	275	8	4600	8.0
				4.5	4.5	250	8	5200	10.6
				4.5	4.5	275	8	5200	9.6
26	55	34	10 Polyurethane	4.5	6.9	250	8	2400	11.6
				4.5	6.9	275	8	2400	12.4
				4.5	5.5	250	8	2400	11.5
				4.5	5.5	275	8	2400	11.8
				4.5	4.5	250	8	2400	11.6
				4.5	4.5	275	8	2400	11.6
27	55	34	10 Kor-Lok 42-3001	4.5	6.5	250	8	5700	10.2
				4.5	6.5	275	8	5600	9.0
				4.5	5.4	250	8	5800	10.0
				4.5	5.4	275	8	5800	9.3
				4.5	4.5	250	8	5700	9.4
				4.5	4.5	275	8	5700	8.9
28	55	34	10 N.S. 78-3730 + 0.5 Cymel 303	4.5	6.5	250	8	4900	10.7
				4.5	5.7	250	8	4900	10.8
				4.5	4.5	250	8	4900	11.6
29	55	34	10 Polyurethane + 0.5 Cymel 303	4.5	6.7	250	8	2000	14.1
				4.5	5.6	250	8	1600	12.9
				4.5	4.4	250	8	1700	13.5
30	55	34	10 N.S. 78-3730 + 0.5 Beetle	4.5	6.2	250	8	4500	9.2
				4.5	5.5	250	8	4600	10.9
				4.5	4.4	250	8	4800	11.3
31	55	34	10 Kor-Lok 42-3001	4.5	7.1	275	8	5000	8.4
				4.5	5.5	275	8	4900	8.1
				4.5	4.5	275	8	5400	9.8
32	55	34	10 Dow 241 + 0.5 Cymel 300	4.5	6.3	250	8	4900	8.4
				4.5	5.5	250	8	4600	9.1
				4.5	4.5	250	8	4300	9.4

<sup>1</sup> 13.4% nitrogen<sup>2</sup> Coupons pressed at room temperature, dried, and repressed<sup>3</sup> Cymel added before Dow 241<sup>4</sup> Vacuum dried

shot tank are consolidated by filtration and the filtrate discarded. Thus a majority of the alum and other water solubles were removed before the felting operation. Starting with Lot No. 14, the filtrate was collected and used to supply part of the water in the 50-gal felting tank.

Other procedure changes are noted in the description of lots No. 14 through 32.

#### **Lot No. 14**

Only Cymel 300 was used as the binding resin in this lot. The resin solution was added to the fibers and agitated for 30 min before the pH was slowly lowered to 3 with p-toluene sulfonic acid. In order to maintain this pH in the felting tank, it was necessary to add hydrochloric acid.

#### **Lot No. 15**

One-half percent Cymel 300 was added to the fibers and agitated for 15 min before Dow latex No. 241 was added. The pH was slowly lowered to 4.5 with alum. One gram (16 wt % of Cymel) of p-toluene sulfonic acid was mixed into the slurry and then the pH was lowered to 3.0 with hydrochloric acid.

#### **Lot No. 16**

One-half percent Cymel 300 and one gram (16 wt % of Cymel) of p-toluene sulfonic acid were mixed together and added to the slurry. The pH was slowly lowered to 4.5 over a 30-min period. National Starch resin No. 78-3730 was added over a 5-min period. After 30 min of agitation, the pH was lowered to 3.0 with hydrochloric acid.

#### **Lot No. 17**

To be most effective, Cymel 300 should cross link the fibers. Thus it appeared logical to deposit the Cymel onto the fiber before the binding resin was deposited. In this batch, the fibers were agitated at a 2 percent solids content while Cymel 300 was slowly added. The pH of the slurry was lowered to 4.5 with alum to precipitate the Cymel onto the fibers. The Dow 241 resin emulsion was then added over a 10-min period. The slurry was aged overnight before diluting and felting the test coupons. One third of the coupons were felted at a pH of 6.8; the felting tank pH was then lowered to 5.8 with alum. The second group of coupons was felted at this pH. The last group of

coupons was felted at a pH of 4.5. Coupons from each group were pressed at either 250° or 275°F. The tensile data obtained are presented in Table 1.

#### Lot No. 18

The recommended curing conditions for Cymel 300 are 320°F at a pH of 3.0 or lower. Although neither of these conditions is acceptable to nitrocellulose processing, these conditions were to be investigated with our system. Lot 18 was prepared using only inert fibers: 77 percent Kraft and 12 percent graphite yarn. The resin system consisted of 10 percent Dow resin 241 and 1/2 percent Cymel 300. Test coupons were felted, pressed, and tested in the usual manner. The coupon densities were lower than usual and the tensile strength very high. Those coupons pressed at 350° or 400°F had significantly less tensile strength. They were also quite brittle as indicated by the low elongation prior to breaking. These coupons were discolored, indicating a degradation of the Kraft fibers. Coupons pressed at a pH of 3.0 had tensile strength similar to those pressed at a pH of 4.5. The lower pH does not affect the Kraft fibers but would be detrimental to nitrocellulose fibers.

#### Lot No. 19

This batch was prepared following the same procedure as in Lot No. 17. Cymel was added to the fibers, the pH was lowered to 4.5, and Dow resin 241 added last. The amount of Cymel was reduced to 0.2 percent. Test coupons were felted at three pH ranges and pressed at either 250° or 275°F. The tensile data are given in Table 1 and also presented in Figure 4. The data indicate that the pH of the felting system must be controlled to optimize the tensile strength. No difference in tensile strength was apparent between coupons pressed at 250°F for 8 min and those pressed at 275°F for 8 min. Tests with thermocouples inserted into the coupons showed that with a press temperature of 250°F, the coupon reached 230°F in 8 min. At a press temperature of 275°F, the coupon reached 260°F in 8 min.

The current practice is to hot press and cure the wet, felted coupon at an initial moisture content of about 60 percent. To determine whether this moisture affected the cross-linking efficiency of Cymel 300, a special mold was fabricated. In this mold, water could be squeezed out of a coupon at room temperature and the coupon thickness maintained by locking the mold. The mold

is placed in a chamber heated to 100°F and evacuated to a pressure of less than 1 Torr. After a specified time, the mold is repositioned between platens heated to 250°F for the final resin cure.

Two coupons of Lot No. 19 were felted at a pH of 7.0 and processed in this manner. One was evacuated for 64 hr, the other for 6 hr. Both were hot pressed at 250°F for 8 min. The drier coupon (64 hr evacuation) had a tensile strength of 2400 psi at a density of 0.8 g/cc. The second coupon had a tensile strength of 3400 psi at a similar density. Since coupons of the same lot pressed in the standard procedure would have a tensile strength of 3200 psi at this density, we concluded that the removal of water before hot pressing does not improve the cross-linking properties of Cymel 300.

#### Lot No. 20

In previous runs the felting (pressing) pH was found to have an effect on the tensile strength. Lot No. 20 was prepared without Cymel to determine if the pH affected only the cross-linking properties of the Cymel resin. Specimens felted between 4.5 and 6.8 pH appeared to have equal tensile strengths indicating that pH control is more important to the Cymel resin curing than to the Dow 241. The tensile strengths of this lot were higher than those obtained in Lot No. 8, which had a similar composition. This could be attributed to a change in the preparation procedure (recycle filtrate) or to the higher felting pH (7.5) of Lot No. 8.

#### Lot No. 21

Cymel 300 precipitates in dilute solution very slowly. In previous batches, 6.6 g of Cymel 300 were added to a slurry of 1200 g of fibers in 60,000 g of water (2%). At this low concentration (90 ppm), deposition of the Cymel onto the fibers appeared questionable. This lot was prepared by adding the Cymel to the thickest slurry that could be mixed: 1200 g of fibers in 18,000 g of water (7%). The slurry was hand mixed for 30 min to distribute the Cymel uniformly. Water was added to dilute the slurry to 2 percent before adding the Dow 241 resin. The pH was lowered to 4.5 over a 30-min period to precipitate the resins. Those coupons felted and pressed at a pH of 5.4 had tensile strengths slightly higher than the other coupons. No major strength improvement was noted for this lot.

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#### Lot No. 22

Zinc chloride is known to have a wetting effect on cellulose fibers. In Lot No. 22, zinc chloride was substituted for the 0.5 percent Lufax 295 normally used as a retention aid. Otherwise, the remainder of the lot was prepared in the same manner as Lot No. 21. Zinc nitrate, a catalyst in melamine polymerization reactions, was added to the system during the felting of the second half of the test coupons. No benefits were evident from adding these chemicals.

#### Lot No. 23

Mr. Bruner of American Cyanamid Co. suggested that Cymel 303 might be a better cross-linking agent in our low solids content slurry. This material is aliquid and dissolves in water more readily. The preparation procedure of Lots 21 and 22 was followed for this batch. Cymel 303 was slowly added to a thick slurry, kept in motion by a dough hook in a Hobart Model AS-200-FT mixer. After 30 min, the pH was lowered to 4.5 and agitation continued for another 30 min. The dough was transferred to the stock preparation tank and sufficient water added to make a 2 percent slurry. The Dow 241 was added and the pH again lowered to 4.5 by adding alum. Since the tensile test data had considerable scatter, no firm conclusions could be made.

#### Lot No. 24

Beetle, a second melamine cross-linking agent supplied by American Cyanamid Co., was tested in this batch. The preparation and felting was identical to that used in Lot No. 23. Tensile tests showed a trend towards higher strengths for coupons felted at the lowest pH.

#### Lot No. 25

For our application, American Cyanamid Co. recommended an acrylic-Cymel combination and supplied sample quantities of this resin system. Difficulty was encountered in precipitating the resin uniformly onto the fibers, and the batch was discarded. If time permits, another attempt will be made after further discussions with American Cyanamid engineers.

#### Lot No. 26

A polyurethane-water emulsion was available in our laboratories. This resin readily deposited onto the fibers when the pH was lowered to 4.5. The

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test coupons had low tensile strengths, which were not affected by the felting pH.

#### **Lot No. 27**

Kor-Lok resin No. 42-3001 was obtained from the National Starch and Chemical Co. Coupons were prepared and tested to provide baseline data for evaluation of the other resin systems. Very good tensile strengths were obtained with this resin. The block diagram in Figure 5 shows each step in the slurry preparation procedure.

#### **Lot No. 28**

After the Lufax 295 had been deposited onto the fibers, excess water was removed and the fibers mixed with the dough hook. Then 1/2 percent Cymel 303 and 1/2 percent p-toluene sulfonic acid were blended into the thick slurry. Alum was slowly added to reduce the pH to 4.4. The solids content of the slurry was lowered to 2 percent and National Starch resin No. 78-3730 added. Additional alum was added to maintain a pH of 4.4.

#### **Lot No. 29**

The procedure of Lot No. 28 was repeated in this lot, except that polyurethane resin was substituted for the National Starch resin.

#### **Lot No. 30**

The procedure of Lot No. 28 was repeated in this lot, except that Beetle was used as the cross-linking agent.

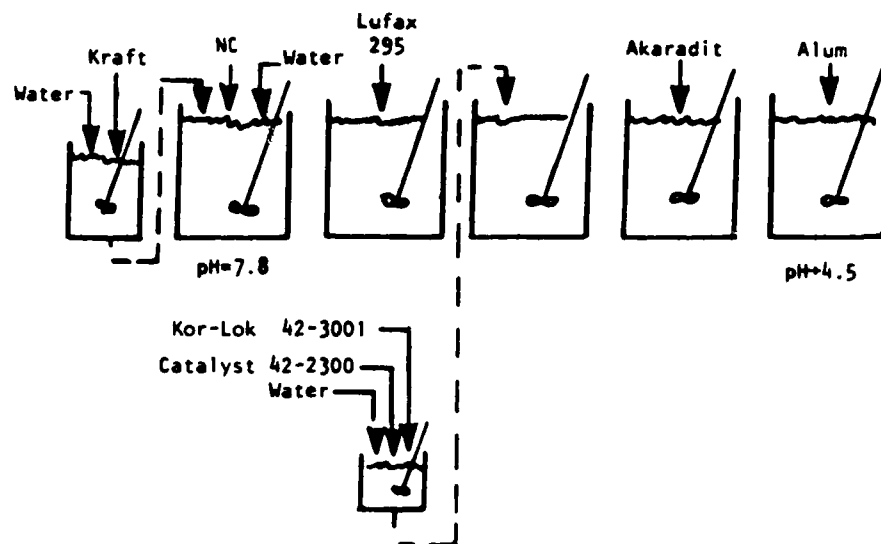
#### **Lot No. 31**

This lot was prepared to duplicate the results of Lot No. 27. The procedure was the same as in Lot No. 27, except that whereas Lot No. 27 was aged over a weekend, Lot No. 31 was only aged overnight.

#### **Lot No. 32**

A thick slurry of Kraft fibers was agitated with 0.05 percent Lufax 295. Cymel 300 and p-toluene sulfonic acid were added over a 60-min period. Nitrocellulose fibers were separately mixed with 0.05 percent Lufax and then blended into a 2 percent slurry with the Cymel-treated Kraft fibers. Dow resin No. 241 was added and the pH lowered with alum in the normal manner.

TIME            0            :15            :30            1:00            1:15            1:30 - 2:00



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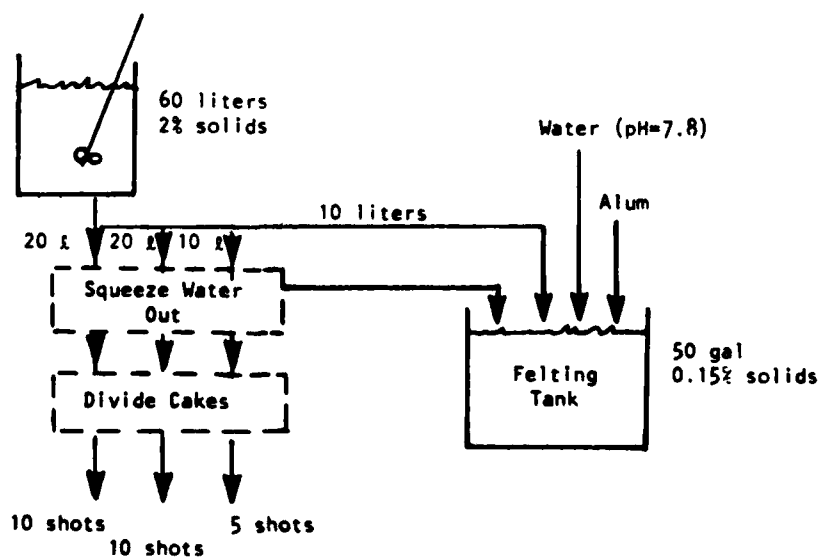


Figure 5. Slurry preparation procedure.

### TECHNIQUE DISCREPANCY

The data appeared to be inconsistent in lots No. 20, 23, and 24. Coupons pressed at the highest pH and at either 250° or 275°F were noticeably weaker than the rest of the coupons of the respective lot. Additional coupons that had been prepared at the suspect conditions were tested and confirmed the previous data. The preparation and felting procedure for each run was reviewed and the reason for the inconsistency discovered.

In charging the stock slurry tank with fibers and water, it was possible for a small quantity of nitrocellulose fibers to be isolated in the discharge line and thus not have resin precipitated onto them. These fibers became part of the initial charge to the felting tank. The first coupons to be felted contained some of these fibers and thus were weaker. Coupons 1 and 2 were the weakest; successive coupons were stronger as the untreated fibers were removed and replaced by treated fibers. Of the first 16 coupons felted at a pH of 6.8, Nos. 1 through 8 were pressed at the existing press temperature, either 250° or 275°F. Coupons No. 9 through 16 were pressed when the press temperature was reset to the other temperature. The stock preparation procedure has been corrected to eliminate this fault.

## OBSERVATIONS AND CONCLUSIONS

Several conclusions can be drawn from the data in Table 1:

- Based on tensile strength, both Dow latex 241 and National Starch resin 78-3730 are excellent binders for combustible cartridge cases.
- Coupons made with Kor-Lok resin 42-3001 had unexpectedly high tensile strength. The reason for the high strength was not evident.
- A small benefit is obtained from the addition of Cymel. The method of contracting the fibers with Cymel is critical and the best technique has yet to be developed. The optimum pressing and curing conditions for maximum cross-linking effect of the Cymel resin have not been established. Conditions must be developed that are compatible with the nitrocellulose fibers.
- Slurry preparation procedures are of prime importance. The sequence of events and the time allotted for each event appear critical. Time was not available to study these factors fully.
- Felting pH is important. An overall trend indicates better results are obtained at a pH of 6.8 or lower. However, the optimum pH is dependent on the resin composition and needs to be investigated more fully for each system.
- Pressing at 300°F for 8 min results in a considerable loss of tensile strength. Since these test coupons were discolored, it can be concluded that partial nitrocellulose decomposition weakened the sample. Similar weakening and discoloration occurred in the coupons pressed at 275°F for 16 min. An 8-min press at either 250° or 275°F produces the same tensile strength. A longer press time at 250°F does not increase the tensile strength.

## RECOMMENDATIONS

We recommend that the following studies be pursued:

- Further study is needed to develop procedures to use the potential cross-linking effect of the Cymel resins. In addition to improving physical strength, the cross-linking of fibers should greatly reduce the detrimental effect of propellant migration.
- The unexpectedly high tensile strengths obtained with the Kor-Lok 42-3001 resin suggest that the slurry preparation procedure could be optimized by a detailed investigation of this resin system.
- Other newly developed resin systems should be evaluated, singly and with cross-linking agents.
- Other cross-linking agents, notably the chromium(III) salts or organotitanate chelating agents, should be investigated.
- Resin evaluation should also be based on stability studies, i.e., compatibility with propellants and combustion properties.
- Cross-linking agents should be investigated with the Kor-Lok resin system.

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